

**Shaped SiO<sub>2</sub> body, method for its production and use**

The invention relates to amorphous porous open-pored shaped SiO<sub>2</sub> bodies, methods for their production and their use.

5           Amorphous porous open-pored shaped SiO<sub>2</sub> bodies are used in many technical fields. Examples which may be mentioned include filter materials, thermal insulation materials or heat shields. Quartz articles of any type can furthermore be produced from amorphous  
10 porous open-pored shaped SiO<sub>2</sub> bodies by means of sintering and/or fusion. Highly shaped SiO<sub>2</sub> bodies may in this case be used, for example, as "preforms" for glass fibers or optical fibers. Crucibles for pulling silicon single crystals can furthermore be produced in  
15 this way.

          In principle, amorphous porous open-pored shaped SiO<sub>2</sub> bodies can be produced by compressing appropriate SiO<sub>2</sub> powders, or by a wet chemical process. In the methods known from ceramics for the compression  
20 of powders, for example cold or hot isostatic pressing methods, it is generally necessary to add binders of an organic type in order to obtain a stable shaped body. These binders need to be dissolved out burnt off in a separate step. This is technically elaborate, expensive  
25 and leads to undesired contamination, which it is absolutely necessary to avoid particularly when producing crucibles for pulling silicon single crystals.

          The preferred way of preparing porous shaped  
30 SiO<sub>2</sub> bodies is therefore the wet chemical procedure. Here, distinction is made between a so-called sol-gel route, in which the amorphous porous open-pored shaped SiO<sub>2</sub> body is produced by hydrolysis and condensation of organosilicon compounds in a solvent, a colloidal sol-gel route, in which SiO<sub>2</sub> particles are furthermore  
35 added to the system, and a so-called slip route, in which SiO<sub>2</sub> particles are dispersed in a solvent and subsequently shaped. The main disadvantage of the sol-

gel route is the low resulting solids content in the shaped body. Specifically in the case of fairly large geometries, this leads to very severe cracking and fracture problems. In the colloidal sol-gel route, a high fill factor of the dispersion is achieved by the addition of  $\text{SiO}_2$  particles, so that the resulting solids content in the shaped body is high. Such a method is described in EP 705797 and in EP 318100. Yet even here, the resulting solids contents are only between 40 and 60% by weight, so that the problem of cracking and fracture is not resolved.

WO 01/17902 describes a method in which a solids content in excess of 80% by weight is achieved by the use of different particle sizes. This leads to a substantially higher strength of the shaped  $\text{SiO}_2$  body, although the production of such a dispersion is very elaborate.

EP 653381 and DE-OS 2218766 disclose a slip casting method in which a dispersion of quartz glass particles is produced in water, and the shaped  $\text{SiO}_2$  body is formed by slow water extraction from a porous mold. Here again, solids contents in excess of 80% by weight are achieved. Owing to the diffusion-dependent water extraction, however, the slip casting method is very time-intensive and viable only for shaped parts with thin walls. This disadvantage can be avoided by the use of a pressure casting method.

As described in EP 1245703 or EP 0196717 B1, for example, a dispersion containing  $\text{SiO}_2$  particles is then poured into a pressure casting mold of a pressure casting machine, and dehydrated via a porous plastic membrane to form the shaped  $\text{SiO}_2$  body.

All known amorphous porous open-pored shaped  $\text{SiO}_2$  bodies have the great disadvantage of being very fragile and susceptible to cracking and fracture in the wet or dried state, that is to say in the state not yet consolidated by heat treatment. The problems of microcrack formation that are known in ceramic production processes, for example during mold release,

drying or handling of the shaped  $\text{SiO}_2$  bodies, also lead very often to tearing or fragmentation of the shaped bodies. This is a great problem particularly whenever large shaped  $\text{SiO}_2$  bodies are involved, for example tubes, rods or crucibles, particularly crucibles for the production of silicon single crystals.

Since the addition of binders very often needs to be avoided when producing highly pure amorphous porous open-pored shaped  $\text{SiO}_2$  bodies for applications in the fiber-optic or semiconductor fields, the problem of cracking is then even more serious.

It was therefore an object of the present invention to provide an amorphous porous open-pored shaped  $\text{SiO}_2$  body with improved crack formation behavior.

It was another object of the present invention to provide a method for the production of the amorphous porous open-pored shaped  $\text{SiO}_2$  body according to the invention.

The first object is achieved by an amorphous porous open-pored shaped  $\text{SiO}_2$  body, which consists of two layers and wherein the layers have an identical structure and composition.

Such a shaped  $\text{SiO}_2$  body is produced by pumping a dispersion containing  $\text{SiO}_2$  particles into a pressure casting mold of a pressure casting machine, in which the dispersion is dehydrated via an inner porous plastic membrane and an outer porous plastic membrane to form the shaped  $\text{SiO}_2$  body.

In this case, the dispersion preferably has a fill factor of amorphous  $\text{SiO}_2$  particles between 10 and 80% by weight, preferably between 50 and 80% by weight, and particularly preferably between 65 and 75% by weight.

Polar or nonpolar organic solvents, for example alcohols, ethers, esters, organic acids, saturated or unsaturated hydrocarbons, or water or mixtures thereof may be used as the dispersant.

Alcohols such as methanol, ethanol, propanol, or acetone or water and mixtures thereof are preferably used. Acetone and water or mixtures thereof are particularly preferably used, and water is more particularly preferably used.

The dispersants described above are particularly preferably used in a highly pure form, a form in which they may be obtained by methods known from the literature, for example, or are commercially available.

When water is used, it is preferable to use specially purified water which has a resistivity  $\geq 18$  megaohm\*cm.

A mineral acid, for example HCl, HF,  $H_3PO_4$ ,  $H_2SO_4$  or silicic acid, or ionogenic additives, for example fluorine salts, are preferably added to the water. The addition of HCl or HF is particularly preferred in this case, and the addition of HF is more particularly preferred. It is also possible to use mixtures of said compounds. A pH of 2 - 7, preferably 3 - 6, should then be adjusted in the dispersion.

As alternative, and likewise preferably, a mineral base may be added to the water, for example  $NH_3$ , NaOH or KOH.  $NH_3$  and NaOH are particularly preferred, and  $NH_3$  is more particularly preferred. It is also possible to use mixtures of said compounds. A pH of 7 - 11, preferably 9 - 10, should then be adjusted.

The pH reduction or increase leads to a stronger shell during the shell formation, so that a more stable shaped  $SiO_2$  body is formed.

The specific density of the  $SiO_2$  particles should preferably be between 1.0 and 2.2 g/cm<sup>3</sup>. It is particularly preferable for the particles to have a specific density of between 1.8 and 2.2 g/cm<sup>3</sup>. It is especially preferably for the particles to have a specific density of between 2.0 and 2.2 g/cm<sup>3</sup>.

$SiO_2$  particles with  $\leq 3$  OH groups per nm<sup>2</sup> on their outer surface are preferred, particularly

preferably  $\leq 2$  OH groups per  $\text{nm}^2$ , and more particularly preferably  $\leq 1$  OH groups per  $\text{nm}^2$ .

5 The  $\text{SiO}_2$  particles should have a particle size distribution with a D50 value between 1 - 200  $\mu\text{m}$ , preferably between 1 - 100  $\mu\text{m}$ , particularly preferably between 10 - 50  $\mu\text{m}$  and more particularly preferably between 10 - 30  $\mu\text{m}$ . A particle distribution which is as narrow as possible is furthermore advantageous.

10  $\text{SiO}_2$  particles with a BET surface area of 0.001  $\text{m}^2/\text{g}$  - 50  $\text{m}^2/\text{g}$  are preferred, particularly preferably 0.001  $\text{m}^2/\text{g}$  - 5  $\text{m}^2/\text{g}$ , more particularly preferably 0.01  $\text{m}^2/\text{g}$  - 0.5  $\text{m}^2/\text{g}$ .

15 The  $\text{SiO}_2$  particles should preferably have a crystalline fraction of at most 1%. They should furthermore preferably have a minimal interaction with the dispersant.

20 Amorphous  $\text{SiO}_2$  particles from various sources have these properties, for example fused (re-sintered) silica and any type of amorphous sintered or compacted  $\text{SiO}_2$ . These are therefore preferably suitable for producing the dispersion according to the invention.

25 A corresponding material can be produced in a manner which is known per se in an oxyhydrogen flame. It is also commercially available, for example under the reference Exelica® from Tokuyama, Japan.

30 So long as the aforementioned criteria are satisfied, it is also possible to use particles from other sources, for example natural quartz, quartz glass sand, vitreous silica, ground quartz glasses or ground quartz glass waste, as well as chemically produced silica glass, for example precipitated silica, highly disperse (fumed) silicas (produced by means of flame pyrolysis), xerogels or aerogels.

35 The amorphous  $\text{SiO}_2$  particles are preferably precipitated silicas, highly disperse silicas, fused silica or compacted  $\text{SiO}_2$  particles, particularly preferably highly disperse silica or fused silica, more particularly preferably fused silica. Mixtures of said

various SiO<sub>2</sub> particles are likewise possible and preferred.

It is furthermore preferable to use amorphous SiO<sub>2</sub> particles with a different particle size distribution. Such amorphous SiO<sub>2</sub> particles are obtained by mixing SiO<sub>2</sub> particles, for example fused of fumed silica with a particle size of 1 - 100 nm, preferably 10 to 50 nm, in an amount of from 1 to 50% by weight, particularly preferably in an amount of from 1 to 30% by weight, more particularly preferably in an amount of from 1 to 10% by weight, with the aforementioned amorphous SiO<sub>2</sub> particles.

The nanoscale SiO<sub>2</sub> particles then function as a kind of inorganic binder between the substantially larger SiO<sub>2</sub> particles, but not as a filler material to achieve a higher fill factor. The SiO<sub>2</sub> particles therefore preferably have a bimodal particle size distribution in the dispersion.

In a preferred embodiment, the particles described above are present in a highly pure form, that is to say with a proportion of atomic impurities, in particular metals  $\leq 300$  ppmw (parts per million by weight), preferably  $\leq 100$  ppmw, particularly preferably  $\leq 10$  ppmw and more particularly preferably  $\leq 1$  ppmw.

In another particular embodiment, the dispersion may also contain metal particles, metal compounds or metal salts. Compounds that are soluble in the dispersant are preferred for this, water-soluble metal salts being particularly preferred.

The additives consisting of metal particles, metal compounds or metal salts may be added before and/or after the dispersion is produced.

When producing the dispersion, the dispersant is prepared and the SiO<sub>2</sub> particles are added slowly, and preferably continuously. The SiO<sub>2</sub> particles may nevertheless be added in a plurality of steps (portion-wise).

The pore size and distribution in the shaped SiO<sub>2</sub> bodies produced from the dispersion can be

adjusted in a controlled way through the selection of the  $\text{SiO}_2$  particle size and the grain sizes.

5 All machines and devices known to the person skilled in the art may be used as dispersing machines. Devices which contain no metal parts that could come in contact with the dispersion are furthermore preferred, in order to avoid metal contamination by abrasion.

10 The dispersing should be carried out at temperatures of between  $0^\circ\text{C}$  and  $50^\circ\text{C}$ , preferably between  $5^\circ\text{C}$  and  $30^\circ\text{C}$ .

The gases possibly contained in the dispersion, for example air, may be removed before and/or during and/or after the dispersing, by means of methods known to the person skilled in the art, for example a vacuum. 15 This is preferably done during and/or after the completed dispersing.

In a homogeneous dispersion produced in this way, no sedimentation of the particles takes place for at least 5 min, preferably for at least 30 min.

20 The dispersion is subsequently transferred into a pressure casting mold of a pressure casting machine, in which the dispersion is dehydrated under pressure via an inner porous plastic membrane and an outer porous plastic membrane to form the shaped  $\text{SiO}_2$  body.

25 The pressure casting mold is filled with the dispersion in a manner known to the person skilled in the art, for example by means of pumping.

The filling may be carried out with any desired pressure, but preferably with pressures between 0.5 and 100 bar, particularly preferably with pressures between 30 5 and 30 bar and more particularly preferably between 5 and 10 bar.

The shell formation is preferably carried out at pressures between 0.5 and 100 bar, particularly preferably with pressures between 5 and 30 and more 35 particularly preferably between 5 and 10 bar.

The shell thicknesses that are formed will be between 1 and 50 mm, preferably between 5 and 30 mm, depending on the intended shaped body.

Between 5 and 90 min are required, depending on the shell thickness, the porous membrane and the applied pressure, in order to form geometrically stable shells.

5           The conversion of the dispersion and the shell formation may be carried out at temperatures of from 0°C up to the boiling point of the dispersant. Temperatures between 20°C and 30°C are preferred.

10           The pressure casting mold consists of two porous membranes, which form a closed intermediate space that corresponds to the intended shaped body shape. There is a corresponding inlet at one or more positions in the membrane, which makes it possible to fill the closed pressure casting mold.

15           The two pressure casting mold parts are held together with an appropriate closing pressure, so that filling and shell formation is possible at the pressures described above. Membranes which have an open porosity of between 5 and 60% by volume, preferably  
20           between 10 and 30% by volume, are preferably used as the porous membranes. The pore size of the membrane may be greater than, less than or equal to the size of the SiO<sub>2</sub> particles being used. It is preferable to use a membrane with a pore size of between 10 nanometers and  
25           100 micrometers, particularly preferably between 100 nanometers and 50 micrometers, more particularly preferably between 100 nanometers and 30 micrometers.

30           Preferably, the porous membrane is completely wettable by the solvent of the dispersion, preferably water, so that uniform shell formation can take place.

35           Any plastic known to the person skilled in the art, which is chemically stable and contains no free residues, in particular no metallic residues, is suitable as a material for the membrane. Plastics which are already used in commercial pressure slip casting are preferably suitable. Polymethacrylates and polymethyl methacrylates are particularly preferred.



The thickness of the porous membrane is dictated by the shape of the shaped article to be produced.

5 The water is extracted from the dispersion on both sides into the porous pressure casting membrane parts. The shell is then formed from the outside inward on both sides. The two layers coalesce at the end of the shell formation. A shell formed in this way still consists of 2 layers. The layers are formed identically  
10 in terms of structural layout and composition. A so-called layer boundary is visible to the eye only on fracture edges of shells (Fig. 1). With a high magnification (for example using a scanning electron microscope) a locally limited minimal density variance  
15 can be seen (a so-called texture, Fig. 2).

The shaped  $\text{SiO}_2$  body is released from the mold after the shell formation by moving apart the two pressure casting mold parts and simultaneously applying compressed air and/or water to the porous membranes.  
20 The compressed air or water detaches the shell from the porous membrane because some of the water that has penetrated into the porous membrane is forced in the opposite direction toward the shell, and a thin water film is formed between the shell on the membrane.

25 The shells formed in this way have a solids content of between 80 and 95% by weight.

The shaped  $\text{SiO}_2$  body released from the mold is dried by means of methods known to the person skilled in the art, for example vacuum drying, drying by means  
30 of hot gases, for example nitrogen and/or air, contact drying or microwave drying. A combination of the individual drying methods is also possible. Drying by means of microwaves is preferred.

35 The drying is carried out at temperatures in the shaped body of between  $25^\circ\text{C}$  and the boiling point of the dispersant in the pores of the shaped body.

The drying times depend on the volume of the shaped body to be dried, the maximum layer thickness,

the dispersant and the pore structure of the shaped body.

Minor shrinkage takes place when the shaped body is dried. The shrinkage depends on the fill factor of the wet shaped body. With a fill factor of 80%, the volume shrinkage is  $\leq 2.5\%$  and the linear shrinkage  $\leq 1.0\%$ . The shrinkage is less with a higher fill factor.

The density of the shaped body according to the invention is between  $1.4 \text{ g/cm}^3$  and  $1.8 \text{ g/cm}^3$ .

Shaped bodies according to the invention, which are produced from a dispersion with a bimodal particle size distribution, have a higher strength than green bodies which have been produced from a dispersion with a monomodal particle size distribution.

In a preferred embodiment, in which all the steps are carried out using highly pure materials, the shaped body has a proportion of atomic impurities, in particular metals,  $\leq 300 \text{ ppmw}$ , preferably  $\leq 100 \text{ ppmw}$ , particularly preferably  $\leq 10 \text{ ppmw}$  and more particularly preferably  $\leq 1 \text{ ppmw}$ .

The shaped body which can be obtained in this way is an amorphous open-pored shaped  $\text{SiO}_2$  body of a size and shape, which consists of two layers that are formed equivalently in terms of the structure and composition.

Surprisingly, it is now been found that if microcracks occur on the inside and/or outside of the shaped body, for example during the mold release, drying or handling, the cracks only propagate as far as a plane where the two layer boundaries of the shaped body meet. The shaped body therefore remains stable and the geometry is preserved (Fig. 3).

Owing to their special properties, the shaped bodies according to the invention can be used in a wide variety of ways, for example as filter materials, thermal insulation materials, heat shields, catalyst support materials, and as "preforms" for glass fibers, optical fibers, optical glasses or quartz articles of any type.

In another particular embodiment, a very wide variety of molecules, materials and substances may be fully or partially added to the porous shaped bodies. Molecules, materials and substances that are catalytically active are preferred. All methods known to the person skilled in the art may be used for this, as described for example in US 5655046.

In a particular embodiment, the shaped bodies obtained in this may also be subjected to sintering. All methods known to the person skilled in the art may be used for this, for example vacuum sintering, zone sintering, arc discharge sintering, plasma or laser sintering, inductive sintering or sintering in a gas atmosphere or gas stream.

In another particular embodiment, the shaped bodies obtained in this way may also be vitrified by means of a CO<sub>2</sub> laser, as described in US-A-2003-0104920, DE-A-10260320 and DE-A-10324440.

The layer structure of the amorphous porous open-pored shaped bodies is lost when they are sintered. If completed sintering is carried out, then there will no longer be any layer structure in the shaped body. Furthermore, no texture will be detectable any longer in the completely sintered silica glass regions (Figs 4 and 5).

In this way, it is possible to produce a 100% amorphous (no cristobalite) sintered shaped silica glass body which has a density of at least 2.15 g/cm<sup>3</sup>, preferably 2.2 g/cm<sup>3</sup>. In a particular embodiment, the sintered shaped silica glass body has no gas inclusions and preferably an OH group concentration  $\leq$  1 ppm. In a particular embodiment, in which all the steps are carried out using highly pure materials, the sintered shaped body has a proportion of atomic impurities, in particular metals,  $\leq$  300 ppmw, preferably  $\leq$  100 ppmw, particularly preferably  $\leq$  10 ppmw and more particularly preferably  $\leq$  1 ppmw. The shaped silica glass bodies produced in this way are suitable in particular for all

applications in which silica glass is used. Preferred fields of application are quartz articles of any type, glass fibers, optical fibers and optical glasses. Highly pure silica glass crucibles for pulling silicon single crystals are a particular preferred area of application.

In another particular embodiment, it is possible for molecules, materials and substances that provide the respective shaped bodies with additional properties to be added to the dispersions and/or the porous shaped bodies and/or the sintered shaped silica glass bodies.

In another particular embodiment, compounds which promote or cause cristobalite formation may be fully or partially added to dispersion and/or the porous shaped bodies. All compounds known to the person skilled in the art that promote and/or cause cristobalite formation may be used for this, as described for example in EP 0753605, US 5053359 or GB 1428788. BaOH and/or aluminum compounds are preferred for this.

After such a shaped body is sintered, crucibles for the crystal pulling of Si single crystals, which have a cristobalite layer on the inside and/or outside, or consist entirely of cristobalite, are in particular obtained. These crucibles are particularly suitable for crystal pulling since they are thermally stable and, for example, contaminate a silicon melt to a lesser extent. Higher yields can thereby be achieved during the crystal pulling.

Fig. 1 shows the layer boundary on a fracture edge of a shaped  $\text{SiO}_2$  body according to the invention.

Fig. 2 shows a scanning electron microscopic image of the texture at the layer boundary in the shaped  $\text{SiO}_2$  body according to the invention.

Fig. 3 shows that cracks which occur in the shaped  $\text{SiO}_2$  body according to the invention end at the layer boundary.

Fig. 4 shows a partially sintered shell. The boundary layer (texture) is still visible.

Fig. 5 shows a cross section through a fully sintered shell. The boundary layer (texture) is no longer detectable.

Fig. 6 shows the production of a shaped body in 14" crucible geometry (Fig. 5), as described in Examples 3 and 4.

The following examples will serve to explain the invention further.

**Example 1:**

Production of an  $\text{SiO}_2$  dispersion

3800 g of double-distilled  $\text{H}_2\text{O}$  were put in a 10 liter plastic beaker. 712 g of fumed silica (highly disperse silica, BET surface area  $200 \text{ m}^2/\text{g}$ , available under the name Wacker HDK® N20 from Wacker-Chemie GmbH, Munich) were firstly stirred in over 30 min with a plastic-coated propeller stirrer. 8188 g of fumed silica (silica with an average particle size of  $30 \mu\text{m}$ ), available under the name Excelica® SE-30 from Tokuyama, were then added portion-wise and dispersed over 30 min.

Following complete dispersing, the dispersion was subjected to a slightly reduced pressure (0.8 bar) for 10 minutes, in order to remove any included air bubbles.

The dispersion produced in this way consisted of 8900 g solids, which corresponds to a solids content of 70% by weight (of which 92% was fused silica and 8% was fumed silica).

**Example 2:**

Production of an  $\text{SiO}_2$  dispersion

2190 g of double-distilled  $\text{H}_2\text{O}$  were put in a 10 liter plastic beaker. 187.8 g of fumed silica (highly disperse silica, BET surface area  $200 \text{ m}^2/\text{g}$ , available under the name Wacker HDK® N20 from Wacker-Chemie GmbH, Munich) were firstly stirred in over 30 min with a plastic-coated propeller stirrer. 4931.2 g of  $\text{SiO}_2$  powder (MKC 100 power from Mitsubishi, ground to a D50

value of 12  $\mu\text{m}$ ) were then added portion-wise and dispersed over 30 min.

Following complete dispersing, the dispersion was subjected to a slightly reduced pressure (0.8 bar) for 10 minutes, in order to remove any included air bubbles.

The dispersion produced in this way consisted of 5119 g solids, which corresponds to a solids content of 70% by weight (with a proportion of 3.5% of fumed silica expressed in terms of the amount of solids).

**Example 3:**

Production of a shaped body in 14" crucible geometry (Fig. 6)

Some of the  $\text{SiO}_2$  dispersion from Example 1 (1) is forced from a storage container (2) with a pressure of 10 bar through a line system (3) between two open-pored plastic membranes made of methyl methacrylate (4+5). The membranes have a porosity of 30% by volume and an average pore radius of 20  $\mu\text{m}$ . The distance between the two membranes allows the formation of a 8 mm thick shell. A closing pressure of 200 bar is applied to the two membranes.

The pressure which is applied to the dispersion forces the majority of the water from the dispersion into the membranes. The  $\text{SiO}_2$  shell is formed. The shell grows from both sides with the membranes toward the middle, until the two shell parts coalesce in the middle.

After the shell formation lasting 45 min has taken place, the pressure in the storage pressure is reduced to a positive pressure of 0 bar. Special air and water lines (6) placed in the membranes make it possible to apply air or water to the formed shaped body (7) through the porous membranes for mold release. The shaped body then detaches from the membranes.

The shaped body is first detached from the outer membrane (5). The inner membrane (4) is then moved upward. The shaped body is now hanging from the inner membrane (4). A form-fit support (8) is

positioned under the shaped body. The shaped body is subsequently lowered onto the support and detached from the inner membrane (4). The inner membrane (4) is then moved back upward.

5           The amorphous open-pored porous shaped body which is produced has a solids content of 89% by weight and a residual water content of 11% by weight. After drying at 90°C for 3 hours, the shaped body is completely dried.

10           **Example 4:**

Production of a shaped body in 14" crucible geometry (Fig. 6)

15           Some of the SiO<sub>2</sub> dispersion from Example 2 (1) is forced from a storage container (2) with a pressure of 5 bar through a line system (3) between two open-pored plastic membranes made of methyl methacrylate (4+5). The membranes have a porosity of 30% by volume and an average pore radius of 20 μm. The distance between the two membranes allows the formation of a 8 mm thick shell.

20           A closing pressure of 60 bar is applied to the two membranes.

25           The pressure which is applied to the dispersion forces the majority of the water from the dispersion into the membranes. The SiO<sub>2</sub> shell is formed. The shell grows from both sides with the membranes toward the middle, until the two shell parts coalesce in the middle.

30           After the shell formation lasting 30 min has taken place, the pressure in the storage pressure is reduced to a positive pressure of 0 bar. Special air and water lines (6) placed in the membranes make it possible to apply air or water to the formed shaped body (7) through the porous membranes for mold release.

35           The shaped body then detaches from the membranes.

          The shaped body is first detached from the outer membrane (5). The inner membrane (4) is then moved upward. The shaped body is now hanging from the inner membrane (4). A form-fit support (8) is

positioned under the shaped body. The shaped body is subsequently lowered onto the support and detached from the inner membrane (4). The inner membrane (4) is then moved back upward.

- 5 The amorphous open-pored porous shaped body which is produced has a solids content of 78% by weight and a residual water content of 22% by weight. After drying at 90°C for 3 hours, the shaped body is completely dried.



## WHAT IS CLAIMED:

1. An amorphous porous open-pored shaped SiO<sub>2</sub> body, which consists of two layers and wherein the layers have an identical structure and composition.

5 2. The shaped SiO<sub>2</sub> body as claimed in claim 1, which has a solids content between 80 and 95% by weight.

3. The shaped SiO<sub>2</sub> body as claimed in claim 1 or 2, which has a density between 1.4 g/cm<sup>3</sup> and 1.8 g/cm<sup>3</sup>.

10 4. The shaped SiO<sub>2</sub> body as claimed in one of claims 1, 2 and 3, which has a shell thickness between 1 and 50 mm.

5. The shaped SiO<sub>2</sub> body as claimed in one of claims 1 to 4, has a proportion of atomic impurities, in particular metals  $\leq$  300 ppmw, preferably  $\leq$  100 ppmw, particularly preferably  $\leq$  10 ppmw and more particularly preferably  $\leq$  1 ppmw.

6. A method for the production of a shaped SiO<sub>2</sub> body as claimed in one of claims 1 to 5, wherein a dispersion containing SiO<sub>2</sub> particles is pumped into a pressure casting mold of a pressure casting machine, in which the dispersion is dehydrated via an inner porous plastic membrane and an outer porous plastic membrane to form the shaped SiO<sub>2</sub> body.

25 7. The method as claimed in claim 6, wherein the pressure casting mold is filled with the dispersion by means of pumping.

8. The method as claimed in claim 6 or 7, wherein the filling is carried out with pressures between 0.5 and 100 bar.

30 9. The method as claimed in one of claims 6 to 8, wherein a shell formation is carried out at pressures between 0.5 and 100 bar.

10. The method as claimed in one of claims 6 to 9, which has a method duration between 5 and 90 min.

35 11. The method as claimed in one of claims 6 to 10, wherein the conversion of the dispersion and the shell formation are carried out at temperatures between 20°C and 30°C.

12. The method as claimed in one of claims 6 to 11, wherein the pressure casting mold consists of two porous membrane parts, which form a closed intermediate space that corresponds to the intended shaped body shape.

13. The method as claimed in one of claims 6 to 12, wherein membranes which have an open porosity between 5 and 60% by volume, preferably between 10 and 30% by volume, are used as the porous membranes.

14. The method as claimed in claim 13, wherein a membrane with a pore size between 10 nanometers and 100 micrometers is used.

15. The method as claimed in one of claims 6 to 14, wherein the dispersion has a fill factor of amorphous SiO<sub>2</sub> particles between 65 and 75% by weight.

16. The method as claimed in one of claims 6 to 15, wherein alcohols such as methanol, ethanol, propanol, or acetone or water and mixtures thereof are used as the dispersant.

17. The method as claimed in claim 16, wherein water which has a resistivity  $\geq 18$  megaohm\*cm is used as the dispersant.

18. The method as claimed in one of claims 6 to 17, wherein the SiO<sub>2</sub> particles have a particle size distribution with a D50 value between 10 - 50  $\mu$ m.

19. The method as claimed in one of claims 6 to 18, wherein the SiO<sub>2</sub> particles have a crystalline fraction of at most 1%.

20. The method as claimed in one of claims 6 to 19, wherein the SiO<sub>2</sub> particles in the dispersion have a bimodal particle size distribution.

21. The method as claimed in one of claims 6 to 20, wherein the shaped SiO<sub>2</sub> body is released from the mold by separating the two pressure casting mold parts and simultaneously applying compressed air and/or water to the porous membranes.

22. The method as claimed in claim 21, wherein the shaped SiO<sub>2</sub> body released from the mold is dried by means of vacuum drying, drying by means of hot gases,

for example nitrogen and/or air, contact drying or microwave drying.

23. Use of a shaped body as claimed in one of claims 1 to 6 as filter materials, thermal insulation materials, heat shields, catalyst support materials and as "preforms" for glass fibers, optical fibers, optical glasses or quartz articles,

**Abstract****Shaped SiO<sub>2</sub> body, method for its production and use**

5 An amorphous porous open-pored shaped SiO<sub>2</sub> body, which consists of two layers and wherein the layers have an identical structure and composition.

Fig. 1: Layer boundary on a fracture edge of a green body shell

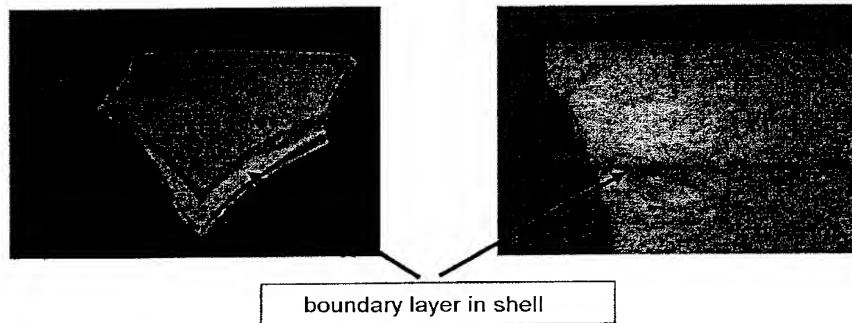


Fig. 2: scanning electron microscopic image of the texture at the layer boundary in the green shell

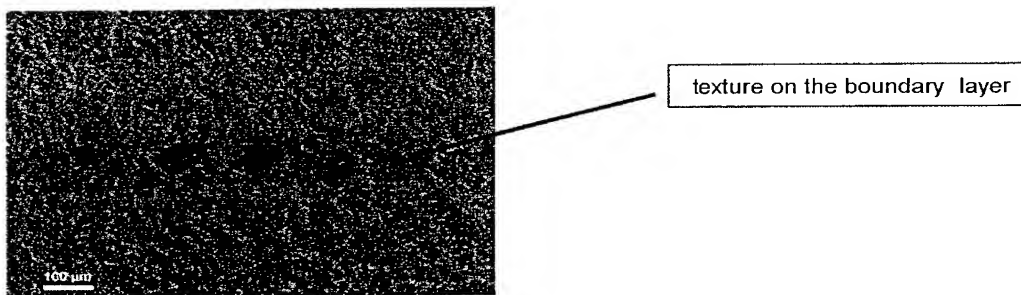
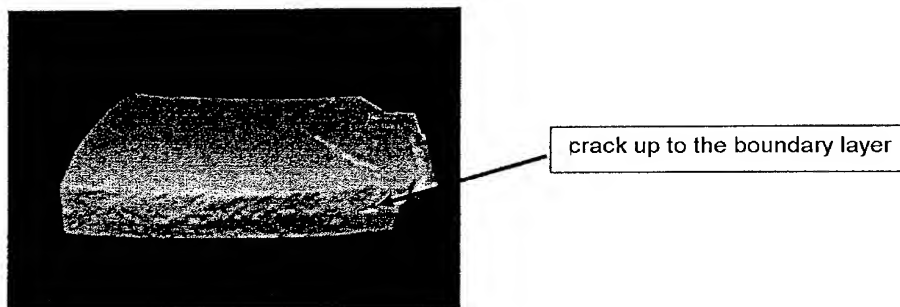


Fig. 3: cracks which occur end at the layer boundary



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Fig. 4: partially sintered shell, boundary layer (texture) still visible

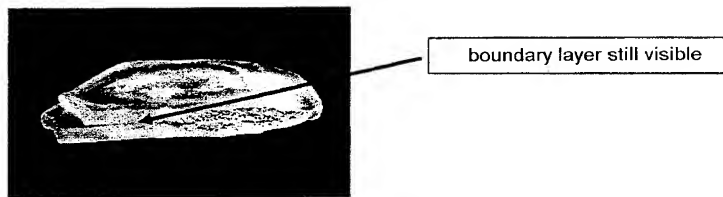


Fig. 5: fully sintered shell, boundary layer (texture) no longer detectable

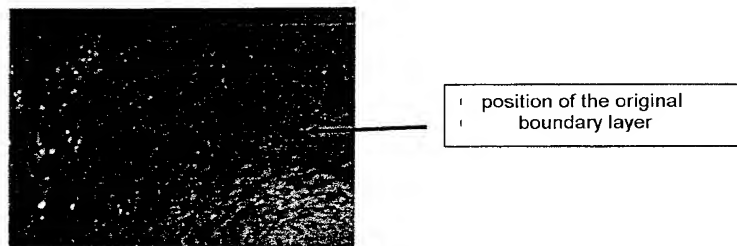


Fig. 6: production of a shaped body in 14" crucible geometry (Fig. 5)

